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AD-A146 661

FUMIGANT DEVELOPMENT FOR KILLING INSECTS ON LETTUCE AND OTHER PRODUCE

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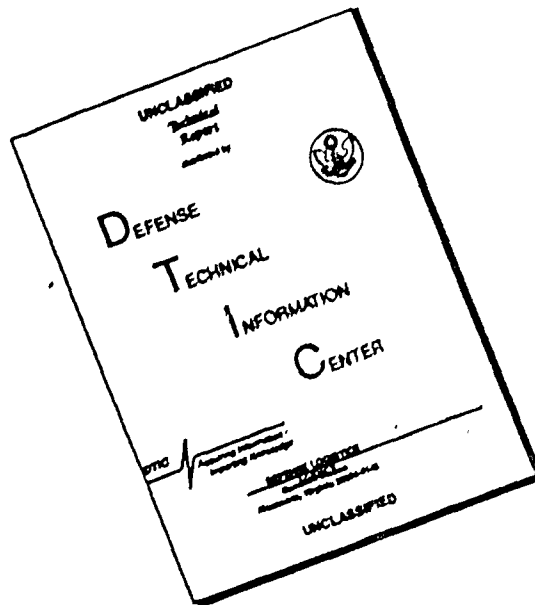
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| REPORT DOCUMENTATION PAGE | | READ INSTRUCTIONS BEFORE COMPLETING FORM |
|--|-------------------------------------|--|
| 1. REPORT NUMBER NATICK/TR-84/046 | 2. GOVT ACCESSION NO. AD-A146661 | 3. RECIPIENT'S CATALOG NUMBER |
| 4. TITLE (and Subtitle) FUMIGANT DEVELOPMENT FOR KILLING INSECTS ON LETTUCE AND OTHER PRODUCE | | 5. TYPE OF REPORT & PERIOD COVERED Final. 1 Oct 80 - 30 Sep 83 Technical Report |
| | | 6. PERFORMING ORG. REPORT NUMBER |
| 7. AUTHOR(s) Ruth Lynn Hooper, John M. Harvey, Garth H. Spitler, Joseph K. Stewart | | 8. CONTRACT OR GRANT NUMBER(s) D83-13 |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS US Army Natick R&D Center Subsistence Protection Branch, Food Technology Division, Food Engineering Lab, STRNC-WTS Natick, MA 01760-5018 | | 10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 1L162724AH99BE AH99BE |
| 11. CONTROLLING OFFICE NAME AND ADDRESS US Army Natick Research and Development Center Kansas Street Natick, MA 01760-5018 | | 12. REPORT DATE April 1984 |
| | | 13. NUMBER OF PAGES 8 |
| 14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) | | 15. SECURITY CLASS. (of this report) Unclassified |
| | | 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE |
| 16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited | | |
| 17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) <div style="text-align: right;">DTIC ELECTE OCT 18 1984 A</div> | | |
| 18. SUPPLEMENTARY NOTES DOD Food RDT&E Program Requirement D83-13 The professional affiliation of John M. Harvey and Garth H. Spitler is Horticultural Crop Research Laboratory, USDA-ARS, Fresno, CA 93747. | | |
| 19. KEY WORDS (Continue on reverse side if necessary and identify by block number) insects fresh vegetables fumigation ethyl formate lettuce aphids vacuum fumigation packaging | | |
| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) When US lettuce shipped to Japan is found at destination to contain live insects, Japanese authorities require fumigation of the entire shipment. Fumigants now used kill the insects but cause extensive damage to the lettuce. This study was conducted to find new fumigants or fumigant combinations and to develop methodology for pre-export fumigation. Twenty-two naturally occurring fumigants were screened for efficacy against commonly found lettuce pests, aphids and lepidopterous larvae, and for phytotoxicity to the lettuce. (OVER) | | |

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The most promising candidate fumigants were acetaldehyde, ethyl formate, ethyl acetate, methyl bromide and a combination of methyl bromide and ethyl acetate. Of these, ethyl formate proved to be the most efficacious (99% kill for aphids) and the least phytotoxic (no detectable damage). Ethyl formate fumigated lettuce was then subjected to residue analyses (none detectable at <0.01 ppm) and taste test panels for flavor changes (no detectable changes). Vacuum fumigation was conducted successfully on a commercial scale with 0.5% ethyl formate. Application for registration of ethyl formate as a fumigant has been submitted to the Environmental Protection Agency (EPA). Pending EPA approval, ethyl formate is recommended for use to fumigate US grown lettuce prior to shipment to Japan.

PREFACE

The objective of this study was to develop an effective and safe treatment against the green peach aphid, Myzus persicae, or other insects that may be present in harvest lettuce. This work was conducted between July 1979 and September 1983. It was supported by US Army Natick R&D Center as Technical Project 1L162724AH99 under approved requirement document DLA 6-1.

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| A1 | |

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TABLE OF CONTENTS

| | Page |
|---------------------------------|------|
| PREFACE | iii |
| INTRODUCTION | 1 |
| MATERIALS AND METHODS | 2 |
| RESULTS AND DISCUSSION | 4 |
| CONCLUSIONS AND RECOMMENDATIONS | 5 |
| REFERENCES | 6 |
| DISTRIBUTION | 7 |

**FUMIGANT DEVELOPMENT FOR KILLING
INSECTS ON LETTUCE AND OTHER PRODUCE**

INTRODUCTION

In 1979, 13.87% of all lettuce shipped by the Defense Logistics Agency to Japan and Okinawa (215,087 pounds of 1,550,718 pounds total) was lost to insect infestation and subsequent fumigation. These losses led to cessation of shipments of head lettuce to Japan and Okinawa from February to May of 1980. From 1980 to the present lesser amounts of lettuce have been shipped per annum. These amounts ranged from a low of 659,595 pounds in 1981 to a high of 867,006 pounds in 1982. Percentages lost to infestation and fumigation have been:

| | |
|------|-----------------------|
| 1980 | 1.3% |
| 1981 | 1.6% |
| 1982 | 4.8% |
| 1983 | 3.8% |
| 1984 | 8.5% (January to May) |

For other fresh fruits and vegetables shipped to Japan and Okinawa from 1981 to present, losses to insect infestation have ranged from less than 0.1% to 0.3% with shipments that have totalled more than 3 million pounds per annum.

When live insects are found on US grown head lettuce shipped to Japan, the Japanese authorities require that the lettuce be fumigated to kill the insects. Fumigants used are hydrocyanic acid or methyl bromide, which kill the insects but severely injure the lettuce. Additionally, when lettuce is fumigated in Japan, each head, which had been film-wrapped at the shipping point, must be unwrapped for the fumigation. The unwrapping is necessary because the fumigation is conducted at normal atmospheric pressure, which prevents the fumigant from passing through the film perforations. The objective of this project is to find new acceptable fumigants and fumigation methods that will kill the insects prior to export without injuring the lettuce or leaving undesirable residues.

Laboratory studies were conducted to determine (1) the efficacy of various naturally occurring volatiles as fumigants for post-harvest control of the green peach aphid (Myzus persicae (Sulzer)), cabbage looper (Trichoplusia ni (Hübner)), corn earworm (Heliothis zea (Brodie)) and tobacco budworm (Heliothis virescens); (2) the phytotoxic responses of iceberg lettuce to these fumigants; (3) possible flavor or odor changes; and (4) residues of fumigants in lettuce.

Large scale commercial tests were conducted as part of the project to determine required methodology for applying ethyl formate, one of the most promising fumigants tested.

MATERIALS AND METHODS

Because the most prevalent insect infesting lettuce in the western United States is the green peach aphid, much of this study focussed on that insect. The infestations by lepidopterous larvae are comparatively rare.

In this study vacuum-fumigation was used because all lettuce packed for export is film-wrapped and packed in cartons. Previous studies show that some fumigants cannot penetrate adequately the perforations in the film-wrap to kill the aphids at normal atmospheric pressure, but that they do penetrate into the lettuce under vacuum fumigation.¹

For this study a series of fumigants was screened for efficacy against both aphids and lepidopterous larvae. Selection criteria for fumigants were that the fumigant was naturally occurring or that the fumigant was commercially available but untested for fumigation against fresh fruits and vegetables. Fumigants were tested at different concentrations, exposure times, combinations, temperatures, and vacuum levels. Initial screening was based on phytotoxicity to the commodity and efficacy as a pesticide. The most promising candidate fumigants were further subjected to sensory evaluations on the fumigated commodity and to residue analyses.

Fumigants that were tested are listed below:

- carbon dioxide
- ethyl acetoacetate
- iso-butyl formate
- iso-propyl acetate
- kodaflex ester alcohol
- methyl acetate
- methyl propyl ketone
- n-butyl acetate
- n-propyl acetate
- ethylene chloride
- ethylene dibromide
- iso-butyl acetate
- methyl aceto-acetate
- methyl acetate
- methyl bromide and ethyl formate
- methyl chloride
- nitro ethane
- acetaldehyde
- ethyl formate
- ethyl acetate
- methyl bromide
- methyl bromide and ethyl acetate

Of the above listed compounds, only the last five were considered sufficiently low in phytotoxicity and high in pesticide efficacy to warrant further testing and evaluation.

Such further testing was similar to that described by Stewart and Aharoni² in 1983 for ethyl formate against green peach aphids in packaged head lettuce. In that study freshly harvested head lettuce was film wrapped and packed in cartons (24 heads per carton). The cartons had two ventilation slots in each side and in each end. The lettuce was held at 2.5°C until the day before fumigation, at which time two heads were unwrapped in two of the three cartons to be fumigated. A radish leaf with about 70 green peach aphids was placed under the third leaf of each unwrapped head. The heads were then rewrapped with the same type of perforated film as used originally. Slit perforations, each 5 mm long and 80 to 90 per sheet, permit moisture to escape from heads during vacuum cooling and provided entrance of the ethyl formate into the heads during vacuum fumigation. Aphids were similarly placed in lettuce heads in two control cartons in each of the three replications.

Immediately after addition of the aphids, the cartons were transferred to a 15°C storage area to simulate harvest time temperature. The lettuce was fumigated the following day. The laboratory vacuum chamber (0.52 m³ capacity) was loaded with three cartons (two with aphids and one filler) of lettuce for each fumigation. The load factor of percent of chamber occupied by the cartons of lettuce was 38%. This is the maximum load factor for most commercial vacuum-cooling chambers.

Vacuum fumigation was performed by first reducing the chamber pressure to 30 mm Hg absolute then introducing the desired dosage of liquid ethyl formate via a pre-placed tube and syringe. Then the absolute pressure was immediately increased to 60 mm Hg. At the end of the fumigation period, the vacuum was broken by introducing air. In order to "air wash,"³ the chamber was again evacuated to 30 mm Hg then air was again introduced until normal atmospheric pressure was attained. Fumigated and control lots were held at 2.5°C for seven days before aphid mortality and lettuce quality were determined.

Fumigant dosages of 0.5%, 1.0%, and 1.5% ethyl formate were tested at a 2.0 hour interval. Then, 0.5% ethyl formate was tested at intervals of 0.5, 1.0, and 1.5 hours. Treatments were repeated in different cultivars of lettuce. Heads of lettuce from the treated and control cartons were rated for general external appearance and defects using the rating scales described by Kader, Lipton and Morris.⁴

Ethyl formate residue analysis was conducted on a Hewlett-Packard 5830A gas chromatograph equipped with a flame ionization detector and a 1.5 m x 4mm ID glass column packed with 80/100 mesh Poropak N. The glass injector insert was packed with a 1 cm to 2 cm length of 3% SE-52 on 80/100 mesh Chromosorb G held in place with quartz wool. The column was maintained at 150°C, the injector at 200°C, and the detector at 225°C. The helium carrier gas flow rate was 35 mL/min.

Commercial-scale vacuum fumigation with ethyl formate against green peach aphid was conducted by Stewart and Mon.⁵ In these tests 480 cartons of palletized lettuce, a 36% load factor, were fumigated in a series of four tests with three replicates each. Lettuce heads were wrapped in perforated polyethylene film. The pressure in the loaded vacuum tube was reduced to 30 mm Hg absolute at which time a dosage of liquid ethyl formate was introduced. Immediately after the ethyl formate was introduced, air was introduced into the tube until the chamber pressure reached 60 mm Hg absolute. Dosages tested were 0.5% and 0.9%. All fumigations were at 1 hour at 60 mm Hg absolute.

RESULTS AND DISCUSSION

In general, for all fumigants tested, aphids, thrips and white flies were much more vulnerable to fumigants than were the lepidopterous larvae. Infestation of lettuce by lepidopterous larvae is a fairly local occurrence and much less common than is infestation by aphids, thrips and white flies.

Perforated film-wrapped heads were as successfully fumigated as naked heads when fumigation was conducted under vacuum. In all tests conducted, the fresher the lettuce, the less the phytotoxic effects from fumigation.

Of 22 fumigants and fumigant combinations that were screened for pesticidal efficacy and phytotoxicity, only 5 were considered to merit further investigation. These 5 were acetaldehyde, ethyl formate, ethyl acetate, methyl bromide, and methyl bromide plus ethyl acetate. Results obtained with these 5 fumigants or combinations are summarized below. Unless otherwise noted, the vacuum chamber was evacuated to 30 mm Hg whereupon the fumigant was introduced, then the pressure was allowed to rise to 60 (or 100) mm Hg for the designated exposure time.

Efficacy and Phytotoxicity of Fumigants

Acetaldehyde: Fumigation at 21°C and at normal atmospheric pressure for 4 hours with 1% acetaldehyde (Aa) in the presence of 50% to 60% CO₂ or 1.5% Aa in 35% CO₂ killed 100% of the green peach aphids on harvested head lettuce without causing injury.^{6,7,8}

Vacuum fumigation with 1.5% Aa at 21°C and at an initial pressure of 30 mm Hg for 2 hours killed 100% of the aphids in lettuce wrapped in perforated film without injuring the lettuce.¹

Ethyl formate: Vacuum (30 mm to 60 mm Hg) fumigation at 15°C for 1 hour with 0.5% ethyl formate (EF) killed 93% to 97% of the aphids in film-wrapped, packed head lettuce without injury to the lettuce.² This dosage did not control the lepidopterous larvae.

Ethyl acetate: Vacuum (30 mm to 60 mm Hg) fumigation at 7.5°C for 2 hours with 0.5%, 1%, or 1.5% ethyl acetate (Ea) killed 100% of the green peach aphids on lettuce, but did not control cabbage looper or corn ear worm. One-half percent Ea at 15°C for 1 hour was effective in controlling aphids in two out of three tests. 0.75% Ea at 15.5°C for 1 hour killed 93% to 99% of the aphids (three tests) and 50% of the cabbage looper (one test) without injury to the lettuce.

Methyl bromide: Vacuum (30 mm to 100 mm Hg) fumigation at 7.5°C for 3 hours with 3g methyl bromide (MB) per cubic meter killed 100% of the cabbage loopers on lettuce without injury. Aphids and corn earworms were only partially controlled.

Methyl bromide + ethyl acetate: Vacuum (30 mm to 100 mm Hg) fumigation at 7.5°C with a combination of 1% Ea and 3g MB/m³ for 4 hours killed 100% of the green peach aphid on lettuce without injury. Ea at 0.5% + 5g MB/m³ for 3 hours at 10°C to 15°C also killed the aphid, as well as the cabbage looper. Neither combination treatment controlled the corn earworm satisfactorily.

Flavor and odor evaluations were conducted on lettuce treated with ethyl formate. An expert panel could not detect any differences in flavor or odor between treated and untreated lettuce.

Residue analyses were conducted on ethyl formate treated lettuce. Ethyl formate residues were not detectable (<0.01 ppm) in treated lettuce. However, trace amounts (0.01 ppm to 0.7 ppm) of ethanol were found in a few samples.⁵

When Stewart and Mon⁵ conducted commercial-scale vacuum fumigation with ethyl formate, replicated tests showed that effective control of the green peach aphid could be accomplished in a chamber normally used to vacuum cool lettuce. Only slight modification of the cooler was required to introduce the fumigant. At a 36% load factor, a dosage of 0.5% ethyl formate at an absolute pressure of 60 mm Hg for 1 hour at 15°C to 20°C controlled 98% to 99% of the aphids in film-wrapped lettuce.

Ethyl formate fumigation applied to Brussels sprouts and broccoli was also effective (99% kill) against green peach aphids and resulted in no detectable phytotoxicity to the vegetable.

In December 1983 application was made through the IR-4 to the Environmental Protection Agency (EPA) for new use registration of ethyl formate as a fumigant. At the time of the publication, EPA registration is still pending.

Estimated cost for ethyl formate fumigation of lettuce is \$1.32 per carton. This cost estimate includes the cost of the chemical at \$0.02 per carton and the use of the cooling chamber for one hour at \$1.30 per carton. Because the cooling chamber is rented for the fumigation process, the cost may be reduced if the fumigation is performed during early morning or evening (nonpeak) hours.

CONCLUSIONS AND RECOMMENDATIONS

Based on the forementioned studies, it is recommended that when EPA registers ethyl formate for use as a fumigant to kill pests on fresh lettuce, ethyl formate fumigation be performed on lettuce shipped to the Orient. Actual application should conform with methods described by Stewart and Mon.⁵ For any fumigation conducted on post-harvest lettuce, the use of very freshly harvested lettuce minimizes the phytotoxic effects of fumigation. The load factor for vacuum fumigation with ethyl formate is 36% to 38%.

This document reports research undertaken at the US Army Natick Research and Development Command and has been assigned No. NATICK/TR-841046 in the series of reports approved for publication.

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